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Conversion of Municipal Sludge to Oil
(Journal Version)

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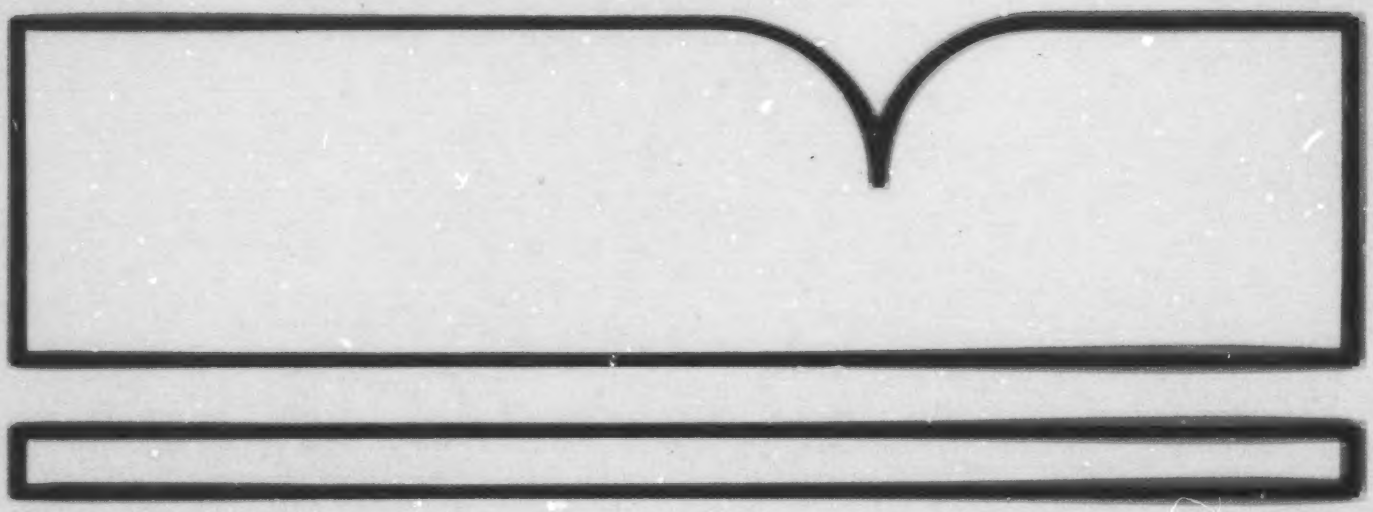

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16. ABSTRACT
Thermal conversion of municipal sludge to oil has been investigated as a viable alternative for ultimate sludge disposal due to generation of energy. The conversion process using water as a solvent was evaluated in a batch mode using primary and secondary municipal sludges. A well defined oil separation procedure was developed in this research. The oil obtained after thermal conversion was dark brown, solid at room temperature and liquid at above 50°C, with an average heat content of 9400 cal/g oil. The increase of oil mass and energy yields as a result of thermal reaction was 73 and 67% in the primary sludge, and 515 and 485% in the secondary sludge, respectively, all with 10.9% volatile solids content. Catalyst addition did not increase the oil mass and energy yields. Data showed that primary sludge in the thermal conversion process.

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Conversion of municipal sludge to oil

Kun M. Lee, Patrick Griffith, Joseph B. Farrell, Atal E. Eralp

Sludge disposal has been a major challenge in the wastewater treatment field. Approximately half of operating and maintenance costs for wastewater treatment plants are incurred by the sludge disposal processes. Current sludge disposal means in the U. S. include incineration, landfilling, ocean disposal, and land spread. All these means have drawbacks; they are either too costly to operate or may create a potential threat to the public health and environment. Therefore, an economical sludge disposal process that ensures protection of public health and environment must be developed.

Conversion of municipal sludge to oil has been investigated as a viable alternative for ultimate sludge disposal in recent years.[1-4] This is because the technology not only eliminates sludge disposal problems, but also generates fuel energy in the form of oil that is transportable and marketable. Current conversion processes, all under development, may be classified into three major categories; pyrolysis,[1,2] the use of oil as a solvent,[3] and the use of water as a solvent.[4] All these processes are operated in an oxygen-free environment.

An oil separation method proved critical for the determination of mass yields.

Pyrolysis has been used to convert organics in municipal refuse and sludge to oil under high temperature, 450° to 800°C, and under pressure close to atmospheric pressure.[5] Recent progress in the pyrolysis process,[1] however, used low operating temperatures (250° to 320°C) with oil yields ranging from 20.2 to 27.4% from a feed sludge of 40 to 50% total solids (TS). The reaction time was less than 2 hours including preheating time.

Young and Sachdev[2] evaluated a modified pyrolysis process[1] to demonstrate oil production from municipal sludge. Dried and finely ground feed sludge was heated in a 2.7-L Pyrex reactor for approximately 3 hours. Oil yields ranged from 12.8 to 28.7% based on TS (the average is 20.4%), and the energy content of oil ranged from 7600 to 9300 cal/g oil (the average is 8636 cal/g oil). They reported that oil yield increased with temperature. However, the yield did not increase significantly above the optimum temperature. At 350°C, the oil yield was 12%, whereas at 400°C, the yield was 24.1%. They also reported that addition of catalysts such as Ni and Co did not increase oil yield. This occurred because of the complex nature of sludge, which may already contain the necessary catalysts.

A process using oil as a solvent was operated under the hydrogen environment at pressures of 100 to 150 atm and temperatures of 300° to 425°C.[3] Anthracene oil and sodium carbonate as a carrier solvent and a catalyst, respectively were added to completely dried and finely ground feed sludge. Oil yields ranged from less than 20 to 50% in a reaction time of 20 minutes or less. However, the investigator concluded that the process was not a viable one because of the high cost of sludge drying, the complex nature of the process, and high ash content of the sludge.

The conversion process using water as a solvent was operated at a temperature of 275° to 350°C in under the hydrogen[3] or nitrogen[4] environment. The operating pressure was mainly caused by water vapor produced at a given temperature. Typically, the maximum operating pressure was less than 140 atm (2000 psi).

Kranich[3] used sludge of approximately 5% TS for a batch operation in a 1-L stirred autoclave. The oil yields were 11 to 14% at 285°C and 27% at 295°C. Thermal reaction time was 15, to 20 minutes at 285° to 295°C, excluding preheating time. Apparent conversion of organics to oil was extremely variable and did not seem to depend on the addition of hydrogen or a catalyst such as Ni and Mo. Most of the time, the oil and asphatens measured in the product were less than those in the feed sludge.

Molton *et al.*[4] used feed sludge of 18.7 to 23.6% TS by centrifuging raw sludge with the aid of polymer. The amount of the sodium carbonate reactant added was 5% by weight of wet feed sludge. Thermal reaction occurred in a continuous flow mode at a sludge feed rate of 720 L/d at an operating temperature, pressure, and reaction time of 275°C, 150 atm, and 90 to 150 minutes, respectively. The oil yields ranged from 7.1 to 24.9% with an average of 15.3%. The oil content in the feed sludge ranged from 9.5 to 20.4% (average 15.9%). Heat content of both oils were 7678 to 8970 cal/g oil (average 8430 cal/g oil). Thus, the oil recovered in the product was slightly less than that in the feed originally. Kranich[3] observed the same results in a batch mode reaction.

Oil separation from the reaction product was quite difficult although no researchers addressed the issue. Low oil recovery may have resulted from an inadequate oil separation method. No clearly defined oil separation procedure has been reported, which is of major importance in practical application of the conversion process. Furthermore, no comparative study between primary sludge and secondary sludge (waste-activated sludge) on the oil yield was conducted. Therefore, the objectives of this research were to determine:

- Whether the process using water as a solvent can convert organics other than oily matter in sludge to oil;
- The methodology of the oil separation procedure;
- The effect of sludges with different oil and grease content and different origin, that is, primary and secondary sludge, on the oil yield.

MATERIALS AND METHODS

A 1-L stirred autoclave made of 316 suspended solids (SS) was used as an experimental reactor. The autoclave rated at

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5000 psig at 345°C (650°F) is located at the U. S. Environmental Protection Agency (EPA) Test and Evaluation facility in Cincinnati, Ohio. Figure 1 shows the batch reactor system used in this study.

Primary sludge from the Little Miami Wastewater Treatment Plant in Cincinnati, Ohio, was gravity thickened or centrifuged, or both, if a higher solids content sludge was needed. Approximately 400 g of wet sludge was used as feed sludge. When catalysts were added, the catalyst dosage was 5% of wet sludge weight. After adding sludge to a cold autoclave, nitrogen gas was used to drive out oxygen from the reactor and then to pressurize it to 100 psig. The initial nitrogen pressure was maintained as an indicator of pressure leakage. The electric heater surrounding the reactor's outer wall supplied needed heat to raise the reactor's temperature which took 1.5 hours. The operating temperature, pressure, reaction time, and stirrer speed were 345°C, 2500 psig, 1 hour, and 500 rpm, respectively. After completion of the reaction, the reactor was cooled down to room temperature, which took 3.5 hours.

A reactor off-gas sample was collected in a gas sampling bag. Reaction product was collected in a glass bottle using a 50-mL wide mouth pipet. The product, still adhering to the reactor's walls and stirrer, was recovered by dissolving it in a fluorocarbon solvent (1,1,2-trichloro-1,2,2-trifluroethane), and collected in a separate glass bottle. All glass sampling bottles were cleaned with solvent.

A new separation method using a fluorocarbon solvent as an extraction solvent was developed in this study. The sample, either reaction product or feed sludge, was adjusted to pH 2.0 using (1 + 1) HCl to convert fatty acid salts into free fatty acids. The free fatty acids are soluble inorganic solvent; thus oil extraction from the sample can be maximized. Approximately 50 g of sample were added to a 200 mL separatory funnel and pH was adjusted to 2.0. Approximately 50 mL of solvent was added into the separatory funnel and the mixture was shaken vigorously for 5 minutes. The solvent became emulsified; however, spinning the funnel reseparated the solvent layer from the sample layer. The solvent layer containing oil was drained and fresh solvent was again added to the separatory funnel. The extraction procedure was repeated three more times.

Solvent was evaporated from the solvent-oil mixture in a water bath of 65°C under open atmosphere. The endpoint of the evaporation was identified as the point at which solvent bubble formation ceased. Water from the water-solids mixture obtained from the separatory funnel was evaporated in a water bath of 100°C. Residue left after evaporation was dried in a drying oven of 103°C. Thus, the oil in this research was defined as a solvent dissolved substance with a boiling point greater than 65°C under open atmosphere. The char was defined as any substance after oil extraction and water evaporation.

Analytical parameters include TS, volatile solids (VS), oil and grease, total organic carbon (TOC), heat of combustion, and oil and off-gas compositions. Analytical methods for TS, VS, and oil and grease followed "Standard Methods."[6] TOC analysis was performed with a TOC analyzer,[7] and heat of combustion was determined in an oxygen bomb calorimeter.[8] Off-gas was analyzed using a GC with a flame ion detector[6] and oil composition was determined using GC/MS with a capillary column.[9]

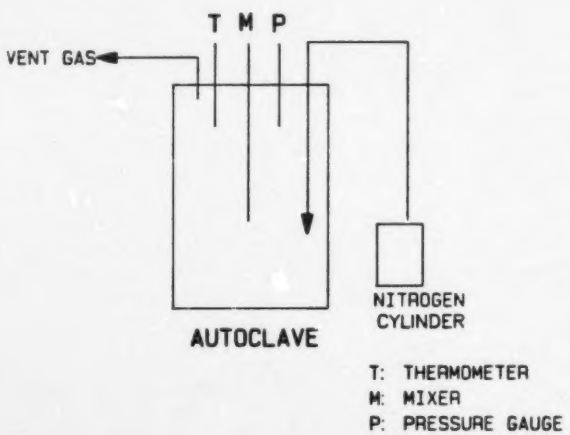

Figure 1—Schematic diagram of the reactor.

RESULTS AND DISCUSSION

The oil from thermal reaction product was dark black, while that from feed sludge was light brown. Both oils are solid at room temperature and liquid at temperatures above 50°C, and are not volatile. The heat content of both oils was similar and averaged 9400 cal/g oil.

The oil yields from thermal reaction products and from feed sludge were measured using 2.65% VS (4% TS) primary sludge collected in July 1986 to determine whether oil was produced from organics other than oily matter in feed sludge. Also, the effect of catalyst addition on the oil yield was investigated. Two different alkaline catalysts, Na_2CO_3 and NaOH, were tested with no catalyst addition as a control.

Figure 2 shows that yields of oil mass and energy increased when sludge was heated under pressure. The oil mass yields defined as grams of oil in the sample divided by grams of feed sludge volatile solids, were 21.8% from feed sludge and 29.5% from reaction product. The oil energy yields, defined as calories in the oil divided by calories in the sludge sample, were 35.5% from feed sludge and 47.4% from reaction product. The relative increase in mass and energy yields of oil as a result of thermal reaction as 35% each. This indicates that oil was produced from organic matter other than oily matter in sludges.

Figure 2 also shows that mass and energy yields of oil among reaction products with different catalyst addition were similar. The average mass yields of oil were 30.9, 29.7, and 28.8% from reaction products without catalyst addition, Na_2CO_3 addition, and NaOH addition, respectively. The average energy yields of oil were 49.4, 47.1, and 46.5%, respectively. This indicates that the addition of either Na_2CO_3 or NaOH as a catalyst into sludge in conversion process does not increase oil mass and energy yields. All the oil yields data have been duplicated as a minimum to ensure high reproducibility. All data show relatively high reproducibility based on the less than 5% coefficient of variation of the oil yield data.

Oil composition analysis by GC/MS shows for each sample two major peaks that were defined by relative concentration and

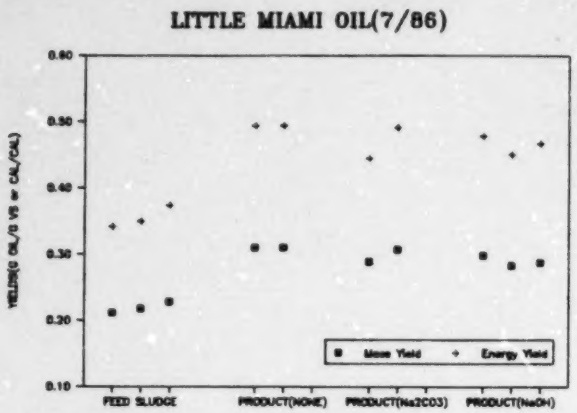

Figure 2—Mass and energy yields of Little Miami Sludge.

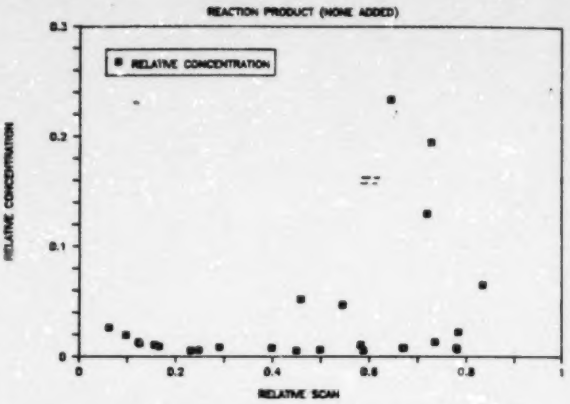

Figure 4—Oil composition chromatogram of reacted Little Miami sludge without catalyst.

relative scan (Figures 3 and 4). The former is the ratio of mass of a compound to the total mass of all compound, and the latter is the ratio of scan of a compound to the total scan, respectively. Of these major compounds shown in Table 1, hexadecanoic acid was present in all reaction products and in the feed sludge with NaOH addition.

A comparison of feed sludge and reaction product oil composition in Figures 3 and 4 shows that without catalyst addition two major peaks match closely with similar relative concentration and scan. The second peaks associated with both oils are the same compound, octadecanoic acid. Similar observations can be made in the case of sludge with NaOH addition: two major peaks matched closely with similar relative concentration and scan. The first peak is the same compound, hexadecanoic acid. Besides these, small peaks produced by both oil samples are quite similar. Thus, oils from feed sludge and thermal reaction products without catalyst and with NaOH are similar in composition.

Oils obtained from Na_2CO_3 addition to feed sludge and reaction product, however, exhibited different composition. Although the relative scans of two peaks produced by both oil samples are close together, the relative concentrations of two peaks associated with reaction product samples were almost twice as great as that from feed sludge. The sum of relative concentration of two peaks was 0.56 in the reaction product, while the sum was 0.30 in the feed sludge.

The oil composition analysis shows similarity between oils from feed sludge and from reaction products without catalyst and with NaOH as demonstrated by the full scan in Figures 3 and 4. However, differences exist in the case of Na_2CO_3 added to sludge (Figure 5). It suggests that Na_2CO_3 may simplify oil composition, although a major portion of oil from the thermal reaction product has been derived from oil already in feed sludge. However, catalyst addition does not increase the oil mass and energy yields. Some research reported that catalyst addition was not needed because sludge already contained numerous catalysts.[5] Others[10] reported no increase of oil yields by adding Na_2CO_3 into Aspen wood which does not contain catalysts. Youngquist *et al.*[11] reported that addition of sodium carbonate to the sludge had no effect on yields and they attributed this to the presence of alkai salt in the sludge in sufficient quantities. Thus, no clear conclusion can be made as to the role and need of catalyst in the sludge to oil process.

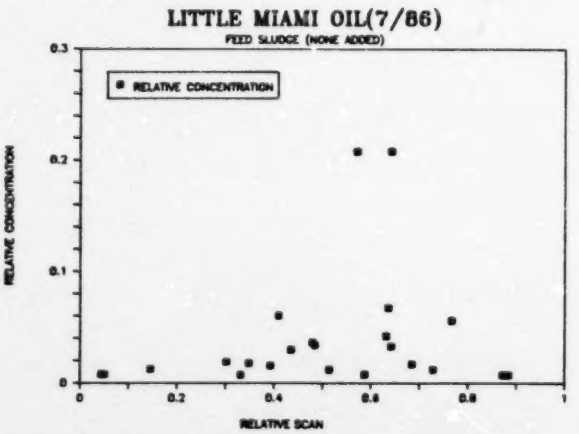

Figure 3—Oil composition chromatogram of unreacted Little Miami feed sludge without catalyst.

Table 1—Major compounds in oil from LMS (7/86).

Item	Compound	Relative concentration	Relative scan
Sludge (None)			
Feed	Unresolved mixture	0.20	0.57
	Octadecanoic acid	0.20	0.64
Reaction	Hexadecanoic acid	0.23	0.64
	Octadecanoic acid	0.19	0.72
Sludge (NaOH)			
Feed	Hexadecanoic acid	0.15	0.61
	Unknown	0.11	0.70
Reaction	Hexadecanoic acid	0.14	0.66
	Unknown	0.13	0.74
Sludge (Na_2CO_3)			
Feed	Carboxylic acid	0.18	0.61
	Decanoic acid	0.12	0.69
Reaction	Hexadecanoic acid	0.30	0.60
	Alcohol	0.26	0.68

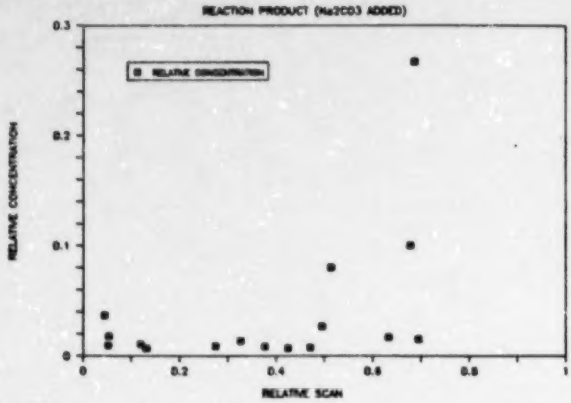

Figure 5—Oil composition chromatogram of reacted Little Miami sludge with Na_2CO_3 addition.

The effect of different sources of sludge with different oil and grease content was evaluated using a fresh batch of primary and secondary (waste activated) sludges from the Little Miami plant in September 1986. Sludges were gravity thickened and centrifuged to simulate mechanically dewatered sludge. For comparison between two different sludges, the VS content of both sludges was equal to 10.9% based on wet sludge weight. The TS content of primary and secondary sludges was 18.5 and 15.7%, respectively. The oil and grease content was 0.234 g/g VS and 0.043 g/g VS for primary and secondary sludges, respectively. Sodium carbonate was added as 25% of TS in the feed sludge.

Three separate thermal reactions and at least duplicate oil extractions from a reaction product were carried out for each sludge. Three separate oil extractions were also performed for feed sludges. Mass balance on solids was attempted by measuring VS of all components in the oil separation steps including oil, char, and sidestream water. Because oil is a desired product in the sludge-to-oil process, all mass and energy yields are considered only mass and energy in the oil.

As shown in Tables 2 and 3, the oil mass and energy yields from primary sludges were 0.181 g oil/g VS and 0.314 cal in oil/cal in feed before thermal reaction and 0.313 g oil/g VS and 0.524 cal in oil/cal in thermal reaction product, respectively. This corresponds to 73 and 67% increase in oil mass and energy yields, respectively, as a result of the thermal reaction. The yields from unreacted secondary sludge were 0.035 g oil/g VS and 0.065 cal in oil/cal in feed and 0.221 g oil/g VS and 0.387 cal in oil/cal in feed after thermal reaction, respectively. This corresponds to 515 and 485% increase in oil mass and energy yields, respectively, as a result of thermal reaction. These results clearly show that oil is produced from organic matters other than oil and grease originally in feed sludge under thermal reaction condition.

Table 2—Mass and energy yields of oil in feed sludge.

	Primary sludge		Secondary sludge	
	Average	Standard	Average	Standard
g oil/g TS	0.1069	0.0054	0.0247	0.0007
g oil/g VS	0.1817	0.0092	0.0359	0.0010
oil cal/feed cal	0.3145	0.0172	0.0659	0.0022

Table 3—Mass and energy yields of oil in reaction product.

	Primary sludge		Secondary sludge	
	Average	Standard	Average	Standard
$\frac{(g\ oil \cdot a) + g\ oil_w}{gTS}$	0.1842	0.0195	0.1519	0.0047
$\frac{(g\ oil \cdot a) + g\ oil_w}{gVS}$	0.3130	0.0331	0.2210	0.0068
$\frac{(oil\ cal \cdot a) + oil_w\ cal}{feed\ cal}$	0.5246	0.0551	0.3877	0.0169

Note—a = ratio of the mass of the recovered product and mass of the extraction sample. Oil_w is oil recovered from the reactor wall.

The GC/MS analysis of oils from primary and secondary sludges shows that two major peaks correspond each to three samples: unreacted primary and secondary feed sludge and the primary sludge reaction product. The oil samples of secondary sludge reaction product produced several peaks of similar magnitude. As shown in Table 4, two major peaks are matching closely between feed and reaction product of the primary sludge. Considering similar relative scan, the hydrocarbon or alcohol compound in oil from reaction product may well be hexadecanoic acid and octadecanoic acid.

As shown in Figure 7, the oil composition of the unreacted secondary feed sludge was quite simple when compared to that of the unreacted primary feed sludge in Figure 6. A simpler oil composition may be caused by consumption of a variety of easily biodegradable oil compounds in the influent wastewater by the microorganisms during the microbial oxidation in the aeration basin. The oil composition of secondary sludge feed and reaction product are different and the difference cannot be explained.

Two major peaks in the oil from the feed secondary sludge

Table 4—Major compounds in oil from LMS (September 1986).

	Compound	Relative concentration	Relative scan
Primary sludge			
Feed	Hexadecanoic acid	0.34	0.45
	Octadecanoic acid	0.22	0.51
Reaction	Hydrocarbon or Alcohol	0.41	0.45
	Hydrocarbon or Alcohol	0.24	0.51
Secondary sludge			
Feed	Hexadecanoic acid	0.36	0.45
	Unknown	0.24	0.51
Reaction	Unknown	0.11	0.04
	Unknown	0.11	0.38
	Unknown	0.11	0.41
	Hydrocarbon or Alcohol	0.19	0.44
	Unknown	0.15	0.46

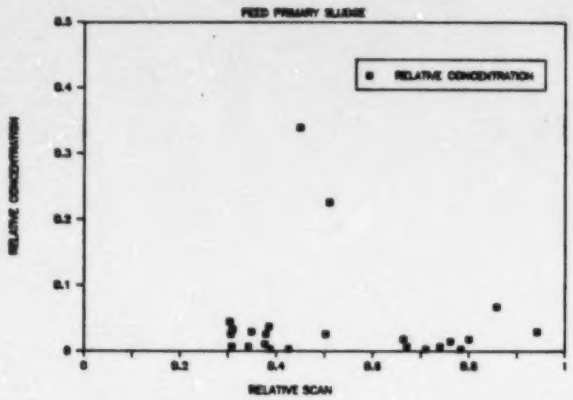

Figure 6—Oil composition of 10.9% VS unreacted primary sludge.

closely match the peaks from the feed primary sludge. The unknown compound in the oil from the feed secondary sludge may be octadecanoic acid considering their similar relative scan. This suggests that both hexadecanoic acid and octadecanoic acid are resistant to microbial oxidation in the aeration basin, and they are major compounds of the primary and secondary sludge oil component.

A comparison of oil composition analyses data between primary sludge of 4% TS (LMS July 1986) in Table 1 and Figures 3 and 4 and primary sludge of 18.5% TS (LMS 9/86) in Table 4 and Figure 6 reveals the same result. This was expected because different TS concentrations in feed sludge from the same source would not affect the oil composition significantly even if taken on different days. The close oil composition analysis result, together with small deviation among oil yields data, strongly indicates that the experimental procedures developed in this study are highly reproducible and analytical quality control was performed well.

A VS mass balance was performed on sludges undergoing thermal reaction. As shown in Figure 8, fractional VS recovery ranges from 0.66 to 0.80 (average 0.712). An incomplete VS recovery is mainly caused by VS loss in the vent gas which is formed during the thermal reaction. The vent gas is of little interest in the sludge-to-oil process because it is close to a non-

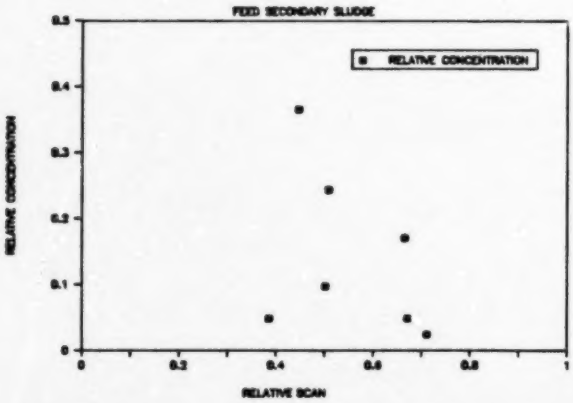

Figure 7—Oil composition of 10.9% VS unreacted secondary sludge.

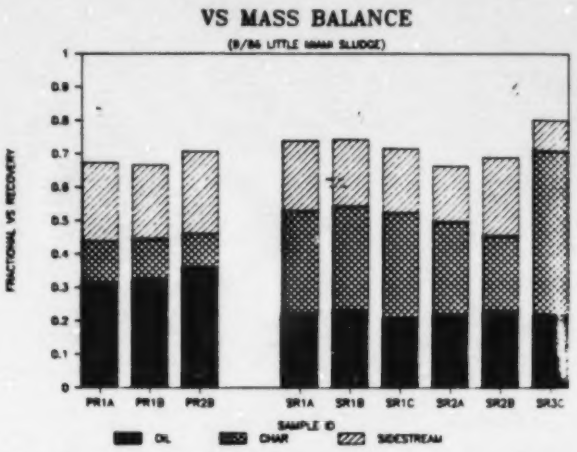

Figure 8—Fractional VS distribution among thermal reaction products.

condensible gas; thus, no quantitive analysis was made on the vent gas except its composition. It is composed mainly of CO_2 and nitrogen. Traces of oxygen were found, but no hydrogen was detected.

Although fractional VS recoveries are similar between reacted primary and secondary sludges, the contribution to the VS mass balance from different components was not the same. The VS contribution from oil, VS component of char, and VS component of sidestream was 48.7, 16.9, and 34.4% to the reacted primary sludge VS and 30.4, 44.3, and 25.4% to the reacted secondary sludge VS, respectively. These results are due to differences in the feed sludge itself. Feed primary sludge contains a significant amount of oil (0.234 g oil/g VS) and VS components are composed of a variety of organic compounds. On the other hand, feed secondary sludge contains small amounts of oil (0.043 g oil/g VS) and VS components are composed mostly of biological solids which are difficult to breakdown under the relatively low reaction temperature used in this study. Thus, primary sludge gives more oil per unit VS applied with less by-product formation than secondary sludge.

TOC content in water phase (sidestream) obtained after oil separation procedure in Table 5 shows that thermal reaction and alkaline pH increased TOC content in sidestream more than that from feed sludge. This was expected because of the vigorous nature of the thermal reaction. Also, chemical bonds of molecules can be destroyed more easily at alkaline pH than at neutral pH. Table 5 also showed that at the VS content of feed primary sludge increased from 2.65 to 10.9%, TOC content in the sidestream from thermal reaction products decreased from 12.8 to 5.4% based on VS content of feed sludge. This is probably because of the relative increase of oil recovery in the product separation process. In other words, any product separation process is subject to an inherent oil recovery loss such as that from attachment to walls of beakers and separatory funnels. Thus, by increasing VS content, more oil can be recovered per unit weight of VS applied. This results in a lower TOC content per VS used in the sidestream. This observation indicates that an efficient recovery of oil during the separation procedure would reduce sidestream strength.

TOC generation from secondary sludge was greater than that from primary sludge, both under conditions with and without

Table 5—TOC in sidestream.

	Primary sludge[a]		Secondary sludge[a]	Primary sludge[b]		Secondary sludge[b]
VS, percent	2.65	10.9	10.9	2.65	10.9	10.9
No Reaction	46.7	13.1	26.8	1 200	1 430	2 890
Reaction						
No Catalyst	110.1	—	—	2 900	—	—
Na_2CO_3	128.3	54.0	100.8	3 400	5 900	10 800
NaOH	182.6	—	—	4 800	—	—

[a] Refers to mg TOC/g VS.
[b] Refers to mg TOC/L wet sludge.

thermal reaction. The TOC mass and concentration from secondary sludge was approximately twice as much as from the primary sludge. This may be caused by the presence of excessive amounts of colloidal matter in secondary sludge compared to primary sludge, which results in a loss of organics during the oil separation procedure. Again, this indicates that an efficient oil recovery during the separation process reduces sidestream strength.

SUMMARY AND CONCLUSION

The feasibility of the conversion process from municipal sludge to oil using water as a solvent was evaluated. Focuses were given on data reproducibility and methodology rather than process optimization. Thus, a batch experiment was conducted using a 1-L stirred autoclave. Effects of thermal reaction, catalyst addition, and VS contents of feed sludges of the oil yields were evaluated. The following conclusions were drawn from this research:

- A well defined product separation procedure was developed;
- Data showed conclusively that oil is a produced from organics other than oily matter in feed sludge by the conversion process;
- The oil mass and energy yields were 0.313 g/g VS and 0.524 cal/cal in the primary sludge, and 0.221 g/g VS and 0.387 cal/cal in the secondary sludge, respectively, under thermal reaction conditions;
- The increase of oil mass and energy yields as a result of thermal reaction was 73 and 67% in the primary sludge, and 515 and 485% in the secondary sludge, respectively;
- Oil composition analysis indicates that the majority of oil in the product may have been derived from oil already in feed sludge;
- Catalyst addition to feed sludge did not increase the oil mass and energy yields;
- Primary sludge produces more oil, energy, and less by-product than secondary sludge in the conversion process; and
- Increases in VS content of feed sludge increased oil recovery and decreased TOC generation in sidestream. This is because of the relative increase in oil recovery as VS content increases.

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